

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: **Brian Gibson, et al.** Examiner: **William K. Cheung**

Application No.: **10/766,430**

Group Art: **1713**

Filing Date: **January 28, 2004**

Attorney Docket No.: **H0003690**

For: **EXTRUDABLE PVC COMPOSITIONS**

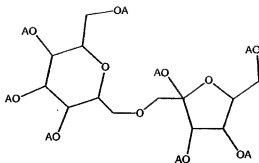
Commissioner for Patents
Alexandria, VA 22313-1450

RULE 132 DECLARATION OF FRED DURRENBERGER

I, Fred Durrenberger, declare and state that:

1. I am an inventor in the above-referenced patent application. I hold a B.S. degree in Chemical Engineering (1978) from University of Missouri-Rolla College. I have been employed by Honeywell International, Inc. and/or its predecessor in interest, the assignee of the application, since about 1992 holding various positions, including my current position as Director of Technology in the Wax and Additives Group. I have extensive experience and familiarity with the development of the extrusion methods and compositions which are the subject of the claims pending in this application.
2. I am familiar with the prosecution history of the above-mentioned patent application and understand that the claims under consideration in this application (as amended as of the date of this filing) are directed generally to a process for extruding a resin-containing composition comprising providing an extrudable mass containing at least one extrudable resin and saccharide ester comprising one or more compounds of in accordance with Formula I as specified in the claims. More particularly, the compounds of Formula I are specified in the claims as follows:

Formula I:



wherein "A" is hydrogen or has the structure of Structure I:

Structure I



wherein "R" is an aliphatic or aromatic moiety of about eight to about 40 carbon atoms, and wherein all of the "A" moieties of at least about 50 wt. % of the compounds of Formula I comprise moieties of Structure I.

3. I am aware that the Patent Office has rejected claims 1 – 18 and 67 of the present application (as previously presented) as being anticipated by US 3,635,856 (Kaneko). I have reviewed both the Office Action and the Kaneko patent and understand the contents of both.
4. I understand the Kaneko patent to relate to non-toxic compositions comprising PVC and stabilizer composed of several additives, including sucrose alkyl ester and polyhydric alcohols. This patent indicates that the purpose of including the sucrose in the composition is because of its interaction with the polyhydric alcohol, namely the formation of eutectic mixtures therewith and also to make certain metal salts included in the composition dispersible in and over the entire body of the PVC composition during the course of mixing. (see col. 3, lines 17 – 30). I also note that this patent teaches that the sucrose alkyl esters may be those having a hydrophile-lipophile balance (HLB) of below 6. However, this patent does not describe the manner by which this balance is calculated and it is difficult in the absence of such a description to assign meaning to this teaching. On the other hand, the patent seems to indicate that the sucrose alkyl esters are preferably diesters or monoesters of fatty acids having an alkyl group with 12 to 18 carbon atoms and mixtures thereof. This description is readily understood to mean that

the esters have either one or two of the hydroxyl groups on the sugar substituted by the ester. I note that, under certain techniques used today to measure what is referred to as HLB balance, such mono- and di-ester sugars would typically have HLB values of greater than 6 (see the chart attached as Exhibit A). Moreover, this patent further indicates that esters resulting from the complete esterification of organic acid have proven to be ineffective to prevent the shaped body to color in the early stages after molding. (col. 3, line 73 – col. 4, line 1). Based on this passage, and other passages (including those mentioned above), I believe that a person skilled in the art at the time our invention was made would believe that highly substituted esters would not have a heat stabilizing effect on PVC compositions. More specifically, I believe that the Kaneko patent, taken as a whole, would teach away from highly substituted esters, included particularly octa-substituted sucrose esters, and instead teaches that mono- and di- substituted sucrose esters should be used.

5. I have tested and/or supervised the testing of three different compositions. The first composition (Sample 1A) is made in accordance with the methods as now claimed in the present application, while the other two compositions (Samples 1B and 1C) are outside the scope of the claims as now amended. The results of these tests are reported in Table I below. Unless otherwise indicated, the materials used to prepare the various compositions were as follows: 1091® polyvinyl chloride resin (resin) from Georgia Gulf; TM281® tin based heat stabilizer (stabilizer) from Rohm & Haas; Rheolub® 165 paraffin wax (paraffin wax) from Honeywell; COAD 10® calcium stearate (calcium lubricant) from Norac; A-C® 629 oxidized polyethylene wax from Honeywell (polymer wax); Paraloid K120N® acrylic processing aid (processing aid) from Rohm & Haas; Paraloid KM334® acrylic impact modifier (impact modifier) from Rohm & Haas; UFT® calcium carbonate from OMYA (calcium carbonate); titanium dioxide (TiO₂), and sucrose stearate obtained from Procter & Gamble. All materials were used as received.
6. The compositions in Table I were prepared by charging the constituent components into a Waring Blender (high intensity mixer) and heating to a temperature in excess of 105°C while operating the mixing blades. When the components were substantially homogeneously blended, the composition was discharged, cooled to about ambient temperature and left to stand for about 24 hours (aging period). At the end of the aging period a sample of about 68g of the composition was placed into a Brabender torque rheometer and subjected to rheometric analysis. Each composition was subjected to rheometric analysis of the fusion and stability time, equilibrium and fusion torque and equilibrium temperature substantially according to ASTM standard testing procedure D2538. The results are presented in Table I below.

Table 1

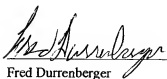
Parameter	Test Sample A	Test Sample B	Test Sample C
Degree of Substitution	At least 70 wt.% Octa <i>Handwritten: 70 wt.%</i>	Estimated 40% mono- and 58% di- (based on Chart attached as Exhibit 1)	Estimated 73% mono- and 27% di- (based on Chart attached as Exhibit 1) <i>Handwritten: 73% mono- and 27% di-</i>
Equilibrium Temp (°C)	212	215	216
Fusion Torque (mg)	3,370	3,900	4,050
Equilibrium Torque (mg)	1,860	1930	1930
Fusion Time (min)	0.78	0.45	0.43
Dynamic Stability time, min.*	14	10	11

*elapsed time between fusion and measurable onset of cross-linking (minutes)

7. As can be seen from Table 1, each of the three PVC resin-containing compositions were prepared substantially the same except that the specific saccharide ester composition used in each was different. More specifically, Test Sample 1A is based on a saccharide ester purchased from Proctor & Gamble under the trade designation Sefose-1618H. A copy of the MSDS sheet for this product is attached as Exhibit B. Test Sample 1B is based on a saccharide ester purchased from Mitsubishi-Kagaku Foods Corporation under the trade designation S-770. A copy of a data sheet for this product is attached as Exhibit C. Test Sample 1C is based on a saccharide ester purchased from Mitsubishi-Kagaku Foods Corporation under the trade designation S-1670. A copy of a data sheet for this product is attached as Exhibit D. The Sefose-1618H used for Sample 1A comprised at least about 70% by weight of octa-substituted sucrose stearate and had a hydrophilic/lipophilic balance (HLB value) calculated by modern methods of less than 1. The S-770 used for Sample 1B is believed to comprise about 40% by weight of mono-substituted sucrose esters, about 58% by weight of di-substituted sucrose esters, and to have a hydrophilic/lipophilic balance (HLB value) calculated by modern methods of about 7. The S-1670 used for Sample 1C is believed to comprise about 73% by weight of mono-substituted sucrose esters, about 27% by weight of di-substituted sucrose esters, and to have a hydrophilic/lipophilic balance (HLB value) calculated by modern methods of about 16. Each composition was subjected to the same Brabender torque rheometer

test and analysis as described above to determine fusion and stability time, equilibrium and fusion torque and equilibrium temperature substantially according to ASTM standard testing procedure D2538.

8. Table 1 illustrates applicants' discovery that the degree of substitution of the saccharide ester in accordance with the present invention has a significant beneficial effect on the fusion characteristics of the shapeable composition, particularly the equilibrium temperature, the fusion and equilibrium torque, fusion time and dynamic stability, with the higher level of substitution exhibiting superior performance than compositions based on lower levels of substitution. This table evidences that compositions comprising saccharide esters having a higher degree of substitution, particularly predominantly octa stearate as in the case of Test Sample A, exhibit properties characteristic of a higher degree of external lubrication than the lower substituted saccharide esters, as evidenced by the increased fusion time and reduced fusion torque. Furthermore, at the equilibrium condition, the melt temperature of the predominantly octa substituted saccharide ester is considerably less than that of the lower substituted compounds, and the equilibrium torque is equal to or lower than that of the lower substituted compounds. Finally the dynamic heat stability appears to be beneficially impacted by the use of compositions based on a high degree of substitution; particularly compounds having predominantly octa substituted saccharides. The Kaneko patent does not in any way suggest this latter finding and in fact would lead a person skilled in the art away from this discovery.
9. I hereby declare that all statements made herein of my knowledge are true and that all statements made on information and belief are believed to be true and further that the statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.


Fred Durrenberger

12/19/06
Date

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2. I am familiar with the prosecution history of the above-mentioned patent application and understand that the claims under consideration in this application (as amended as of the date of this filing).
3. I am aware that the Patent Office has rejected claims 1 – 18 and 67 of the present application (as previously presented) as being anticipated by US 3,635,856 (Kaneko) "as affirmed by" the Mitsubishi-Kagaku internet publication entitled "Introduction of Sugar Esters" (hereinafter "Mitsubishi"). I have reviewed both the Office Action, the Kaneko patent and the Mitsubishi publication, and I generally understand the subject matter of these.
4. It appears that the Examiner has interpreted the statement in Kaneko relating to a material having an HLB below 6.0 as inherently teaching a composition in accordance with the present invention. Such a position is technically flawed. In general, there is a serious question about what information, if any, can be derived with certainty about the

material in Kaneko based on a reported HLB value since there is no indication of the specifications and procedures used to obtain the reported HLB data. For this reason alone, it is submitted that it is not reasonable to conclude that any degree of substitution is "inherent" in the disclosure of this patent. Furthermore, even if the measurement had been reported as being conducted pursuant to known and well-established procedures, such a measurement cannot be considered as a complete and accurate characterization of the degree of substitution of the many compounds that may be included in a given composition. In general, it is not possible to determine from any non-zero HLB value alone the concentrations of the various compounds which may be present in a mixture of compounds having varying degrees of ester substitution. Thus, two compositions having similar HLB values can frequently have substantially different distributions of compounds, particularly as it relates to the relative concentration of compounds having different degrees of ester substitution.

5. I tested and/or supervised the testing of a composition believed to be representative of the material of the type described in the first row of the Mitsubishi publication (HLB =1). The material was obtained from Mitsubishi under the Mitsubishi trade designation "S 170." The results of testing on this material is presented in the Table below as Sample S 170. I also tested or had tested a first composition (Sample 1) made in accordance with the methods as now claimed in the present application. In such a sample, the estimated HLB value would have an HLB value of much less than 1. The results of these tests are reported in Table 1 below:

TABLE 1

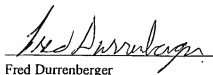
Parameter	Test Sample A	Test Sample S 170
Estimated HLB	<<1	1
Degree of Substitution	At least 70 wt.% Octa	Estimated approximately 10% octa-substituted, 90% of substitution hepta or lower
Equilibrium Temp (°C)	212	215
Fusion Torque (mg)	3,370	3500
Equilibrium Torque (mg)	1,860	1904

Parameter	Test Sample A	Test Sample S 170
Fusion Time (min)	47.5	42
Dynamic Stability time, min.*	13.28	9:33

*elapsed time between fusion and measurable onset of cross-linking

The results of this test work illustrate one important benefit of preferred embodiments of the claimed invention. The dynamic stability of a shapeable composition can be an important measure of the ability of the composition to undergo processing without disadvantageous degradation and cross-linking. As reported in the above table, the compositions in accordance with the present invention as now claimed demonstrate a dramatically superior dynamic stability value compared to Sample S 170. This is generally a highly desirable result.

6. I hereby declare that all statements made herein of my knowledge are true and that all statements made on information and belief are believed to be true and further that the statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.


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